

## SPECIFICATION

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## COMPLETE SPECIFICATION.

## Manufacture of Solid Stable Diazo-azo-salts.

We, I. G. FARBENINDUSTRIE AKTIEN-GESELLSCHAFT, a Joint Stock Company organised according to the laws of Germany, of Frankfurt a/Main, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

10 This invention relates to the manufacture of solid stable diazo-azo-salts by adding to the aqueous solution of diazotised ortho-aminoazotoluene or meta-aminoazotoluene, sulphuric acid sufficient to form the acid diazonium sulphate, and separating the solid diazoazo-salt, thus obtained.

15 Specification No. 1645 of 1896 describes a process of preparing stable zinc chloride double salts of diazo- or tetrazo-compounds of amino-azo-bodies. Example 1 of that Specification describes the manufacture of the zinc chloride double salt of diazotised amino-azo-benzene. When the said Example is applied to the preparation of the zinc chloride double salt of ortho-aminoazotoluene, the salt, thus obtained, possesses an insufficient stability.

20 The diazonium sulphate of ortho-aminoazotoluene, which may easily be precipitated from the aqueous solution, possesses a stability far superior to that of the diazonium zinc chloride double salt and affords diazo-preparations of ortho-aminoazotoluene which are very stable and fit for storing.

25 Even after a storage of 30 days at a temperature of 40° C., the stability of the diazo-salt which has been adjusted to a strength of 20 per cent. of base by means of partly dehydrated aluminium sulphate and anhydrous sodium sulphate, amounts to 90 per cent. whereas the zinc chloride double salt is completely decomposed in the same circumstances. The stability of the corresponding cadmium chloride double salt (cf. Specification No. 294,248) is somewhat better than that of the zinc chloride double salt but does not by far amount to that of the diazonium sulphate.

[Price 1/-]

The diazo-salts from ortho-aminoazotoluene, obtainable according to the process described in German Specification No. 94,280 by precipitating the solutions with naphthalene disulphonic acids, are too sparingly soluble and, therefore, unfit for the preparation of dyestuffs on the fibre.

Furthermore, we have found that the diazo-compound from meta-aminoazotoluene may also be precipitated as acid diazonium sulphate and in this form has a stability essentially better than that of the corresponding zinc chloride double salt prepared according to the process of Specification No. 1645 of 1896. The stability of this diazo-salt which has been adjusted to a strength of 20 per cent. of base by means of partly dehydrated aluminium sulphate (see Specification No. 246,870, page 1, line 25) and anhydrous sodium sulphate, amounts after a storage of 30 days at a temperature of 40° C. even to 80 per cent., whereas the zinc chloride double salt under these conditions is completely decomposed. The stability of the corresponding cadmium chloride double salt (cf. Specification No. 294,248) is somewhat better than that of the zinc chloride double salt but does not by far amount to that of the diazonium sulphate.

The following Examples illustrate the invention, the parts being by weight:—

1) 225 parts of ortho-aminoazotoluene are introduced into 4500 parts of water, while well stirring, and 345 parts of hydrochloric acid of 32.1 per cent. strength are added; the whole is stirred thoroughly and cooled to 10° C. Thereupon, there are gradually run in at about 10° C., 360 parts of a sodium nitrite solution of 20 per cent. strength so that no nitrite is lost. When the diazotisation is finished, the solution is filtered and 250 parts of sulphuric acid of 78 per cent. strength are gradually run in drop by drop. The acid diazonium sulphate of diazotised ortho-aminoazotoluene, is precipitated in part after stirring for a short time. The precipitation is completed by

strewing in 500 parts of anhydrous sodium sulphate. The solution is cooled to  $+5^{\circ}\text{C}$ . and stirred for about 1 hour. The crystalline precipitate is filtered with suction and freed as far as possible from the adherent mother liquor by pressing; thereupon, it is diluted by about its own weight of anhydrous sodium sulphate, for instance, by 320 parts, and dried at  $40^{\circ}\text{C}$ .— $50^{\circ}\text{C}$ . by repeatedly turning it. There is thus obtained the acid diazonium sulphate in the form of a dry reddish-brown powder.

It may be put on the market in this form or may be mixed with a suitable acid binding agent, such as, for instance, anhydrous sodium carbonate or dry borax. There may also be added to the diazo-salt other dyeing assistants such as, for instance, partly dehydrated aluminium sulphate. Thus, for instance, 125 parts of dry borax of the formula  $\text{Na}_2\text{B}_4\text{O}_{10}\cdot 2\text{H}_2\text{O}$  and 200 parts of partly dehydrated aluminium sulphate may be added to the diazo-salt, above described.

2.) 275.8 parts of the hydrochloride of meta-aminoazotoluene of 94.8 per cent. strength are introduced, while well stirring, into 4100 parts of water, and 94.2 parts of sulphuric acid of 78 per cent. strength are added. By external cooling, the temperature of the mixture is brought to  $3^{\circ}\text{C}$ .— $5^{\circ}\text{C}$ . Thereupon, 750 parts of a sodium nitrite solution cooled to  $0^{\circ}\text{C}$ .— $5^{\circ}\text{C}$ . and corresponding with 75 parts of pure sodium nitrite, are introduced under the surface and the whole is stirred until the diazotisation is finished and the liquor still shows a nitrite reaction. Thereupon, 408 parts of sulphuric acid of 78 per cent. strength are run into the liquor which, if necessary, has been clarified and filtered; the diazonium sulphate is thus precipitated in the form of small crystals. The precipitation is completed by introducing through a sieve 366 parts of anhydrous sodium sulphate. Thereupon, the whole is stirred

for 1 hour at a temperature of the liquor of  $4^{\circ}\text{C}$ .— $5^{\circ}\text{C}$ .

The diazonium sulphate obtained is filtered by suction, squeezed, well mixed with 196 parts of anhydrous sodium sulphate and dried at temperatures not higher than  $40^{\circ}\text{C}$ .

The salt may be put on the market in this form or may be mixed with partly dehydrated borax and other dyeing assistants, such as, for instance, aluminium sulphate, for the purpose of removing adherent sulphuric acid.

Thus, for instance, 100 parts of dry borax of the formula  $\text{Na}_2\text{B}_4\text{O}_{10}\cdot 2\text{H}_2\text{O}$  and 208 parts of partly dehydrated aluminium sulphate may be added to the diazo-salt, above described.

The diazo-salt obtained is a brownish-red powder of very good stability.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. The manufacture of solid stable diazo-azo-salts by adding to the aqueous solution of diazotised ortho-aminoazotoluene or diazotised meta-aminoazotoluene, sulphuric acid sufficient to form the acid diazonium sulphate, separating the solid diazo-azo-salt, thus obtained, and mixing it, if necessary, with a diluent or dyeing assistant.

2. The manufacture of solid stable diazo-azo-salts substantially as described with reference to either of the Examples herein.

3. Solid stable diazo-azo-salts, when prepared or produced by the process of manufacture particularly described and ascertained or by any process which is an obvious chemical equivalent thereof.

Dated this 19th day of May, 1933.

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